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STUDIES OF ALKALI METAL CORROSION ON MATERIALS FOR ADVANCED SPACE POWER SYSTEMS

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For Quarter Ending September 26, 1964

By
R.W. HARRISON

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STUDIES OF ALKALI METAL CORROSION ON
MATERIALS FOR ADVANCED SPACE POWER SYSTEMS

QUARTERLY PROGRESS REPORT 1

Covering the Period
June 26, 1964 to September 26, 1964

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I. INTRODUCTION

This report covers the initial period of a capsule program, extending from June 26, 1964 to September 26, 1964, to examine the influence of stress on the corrosion behavior of an advanced refractory alloy in potassium (Task I) and to investigate corrosion mass transfer effects in a stainless steel-columbium alloy-potassium system (Task II).

Task I

While there is considerable evidence that refractory alloys have excellent corrosion resistance to potassium, there are few experiments which describe the possible effects of stress on corrosion when the stress is sufficiently large to produce substantial amounts of creep during the test. It is appropriate for comparative purposes to study an advanced refractory alloy which has demonstrated excellent corrosion resistance to refluxing potassium during long-time exposures conducted at relatively low stresses at 2000°F. In this regard, D-43 columbium base alloy, in the form of welded capsules, has been tested in potassium under refluxing conditions for periods of 5,000 and 10,000 hours at temperatures on the order of 2000°F⁽¹⁾ and has been selected for inclusion in this program.

The D-43 alloy reflux capsules shall be tested under conditions which resulted in about 5 to 10% strain during a 500- to 2,000-hour exposure period in the 2000° to 2200°F temperature range. The capsule wall shall be reduced in the potassium liquid region and in the vapor condensing region to provide gauge sections where the extent of creep can be measured. Moderate temperature adjustments can be made during the test to achieve the desired strain-time conditions.

Task II

The use of stainless steel, rather than refractory alloys, for power plant radiator construction and for the lower temperature portion of experimental facilities would constitute considerable material and fabrication cost savings. However, in bimetallic systems incorporating refractory alloys and stainless steels, the major uncertainty and limitation arises from the mass transfer of interstitial elements from the stainless steel to the refractory alloys.

It is well established that the carbon and nitrogen transfer from Type 316SS to Cb-1Zr alloy at temperatures near 1500°F.⁽²⁾ While some important aspects of this mass transfer behavior have been examined, several critical details require additional investigation. There is a need to define acceptable time and temperature conditions of operation in terms of maintaining satisfactory performance of the refractory alloys, such as Cb-1Zr alloy. Also, there are certain metallurgical aspects of this behavior which should be investigated in an effort to eliminate or reduce the mass transfer rate. In the latter category, it is most appropriate to consider the stabilization of carbon and nitrogen in the stainless steel by the addition of metallic elements which form carbides and nitrides of high thermodynamic stability. Commercially available, titanium stabilized, Type 321SS is one such alloy. A comparative investigation of this alloy and Type 316SS should indicate the ability of the titanium addition to reduce or eliminate interstitial mass transfer in a stainless steel-Cb-1Zr alloy bimetallic system. Columbium-1% zirconium alloy specimens will be exposed to liquid potassium in Type 321SS and Type 316SS capsules for 1,000 hours at 1400°F under isothermal conditions to evaluate this premise.

II. SUMMARY

During the first quarter of this program, the topics abstracted below were covered. The results are interpretatively presented in this report.

Task I - Stress Corrosion Reflux Capsule Tests

A literature search and data compilation of the properties of D-43 alloy were performed.

The D-43 alloy bar to be used in the fabrication of the reflux capsules was ordered.

Design of the strain measurement and other auxiliary equipment was completed and detailed drawings were sent to the NASA Technical Manager for approval. Subsequently, all the component parts were ordered.

Stress calculations for the design of the D-43 alloy capsules were completed and the capsule design was initiated.

Task II - Bimetallic Isothermal Capsule Tests

Type 321SS and Type 316SS pipe to be used in the fabrication of the test capsules was ordered and received. Evaluation of these materials and the Cb-1Zr alloy sheet to be used for the fabrication of test specimens was initiated.

Assembly of the test facility was initiated.

III. TASK I - STRESS CORROSION REFLUX CAPSULE TESTS

A. Material Procurement

The D-43 alloy capsules for Task I will be fabricated from 1-1/4-inch diameter bar stock. The decision to purchase bar in place of tubing was made as a result of substantially higher price quotations and long delivery times that were received from the vendors for D-43 tubing and the greater certainty of achieving the desired grain size in the bar. In addition, by electric discharge machining a core from the bar, material of the same metallurgical morphology is made available for pre-test evaluation. Delivery of the bar is expected November 4, 1964.

B. Capsule Design

In order to control the amount of strain that is induced in the capsule wall during the test, it was necessary to establish the relationship between uniaxial creep strain, as determined by a simple tensile test, and the diametral change obtained in pressurized, potassium-filled, thin-walled tubes. If the ratio of principal stresses and their directions remain constant with time, and the test is performed isothermally, the equation for the change in diameter is:

$$\Delta d = \left[\frac{2pr^2}{t} \right] \cdot \left[\frac{1 - \frac{\nu}{2}}{E} + \frac{3}{4} \frac{\epsilon_{ec}}{\sigma_e} \right] \quad (1)$$

where:

$$\sigma_e = \frac{\sqrt{3}}{2} \cdot \frac{pr}{t} \quad (2)$$

ϵ_{ec} = uniaxial creep strain at any instant of time corresponding to an effective stress σ_e .

r = mean tube radius

ν = Poissons ratio

d = tube diameter at the neutral axis

p = internal pressure

E = Young's modulus

t = tube thickness

This equation excludes thermal strain but includes both the elastic and creep effects; it represents the change in diameter as measured while the tube is still pressurized.

Selection of the starting diameter of the D-43 alloy capsules to be used in these tests was based on the following parameters:

1. Creep strength of D-43 alloy at the test temperature.
2. Desired total uniaxial strain in the capsule wall.
3. Vapor pressure of potassium at the test temperature.
4. Minimum wall thickness of the reduced section. A minimum number of 10 grains across the wall is required.

In order to achieve a total uniaxial strain of 5 to 10% and maintain a test temperature in the 2000° to 2200°F range, a minimum wall thickness of at least 0.020-inch and a reasonable tube diameter, a 1-1/4-inch diameter Schedule 40 pipe size was selected. This particular pipe size was established using the estimated average 1% creep properties of D-43 alloy, plotted in Figure 1, in conjunction with the vapor pressure data of potassium, shown in Figure 2, and equation (2). From these data, a plot of the inside tube radius vs the test temperature to obtain 1% creep in 300 hours can be made. This plot is shown in Figure 3 for various wall thicknesses with the 1-1/4-inch diameter pipe size indicated. The parameter of 1% strain in 300 hours was an arbitrary selection based on the available creep data for D-43 alloy. Evaluation of the creep properties of the D-43 alloy bar to be used for the fabrication of the actual capsules will be performed to determine steady state, second stage creep rates. These data will be projected to give the parameters necessary to achieve the desired 5 to 10% strain in 1,000 hours. Any necessary modifications in the capsule design with respect to the reduced wall thickness will be made at that time.

The reduced wall sections of the capsule are being designed such that the induced stress is attenuated by use of a machined fillet. This technique was developed by Grodzinski⁽³⁾ to reduce stress concentrations in shafts. A sketch of the refluxing capsule showing the reduced wall sections in the vapor and liquid zones is shown in Figure 4. Details of the gauge section will be reported when the design is completed.

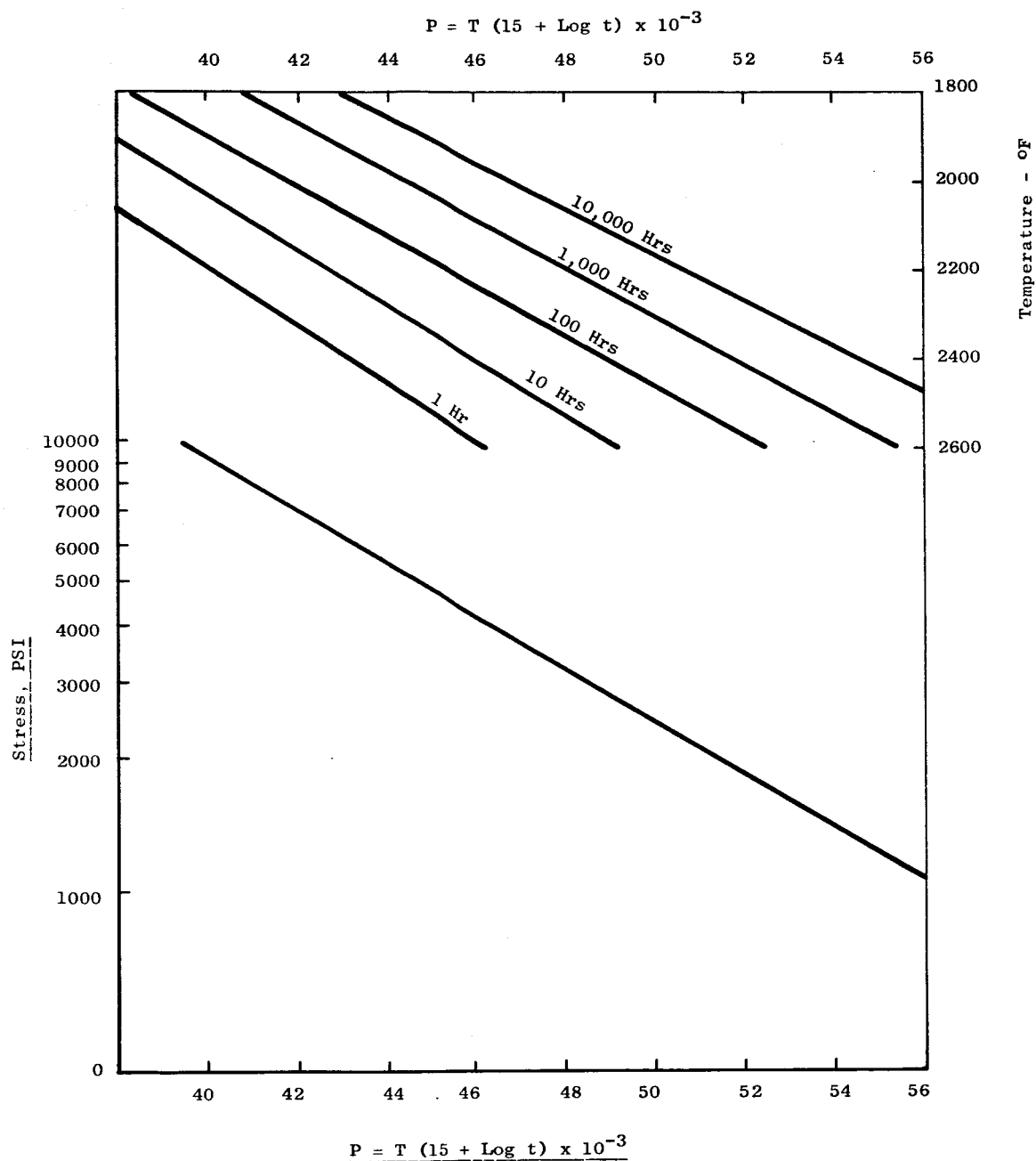
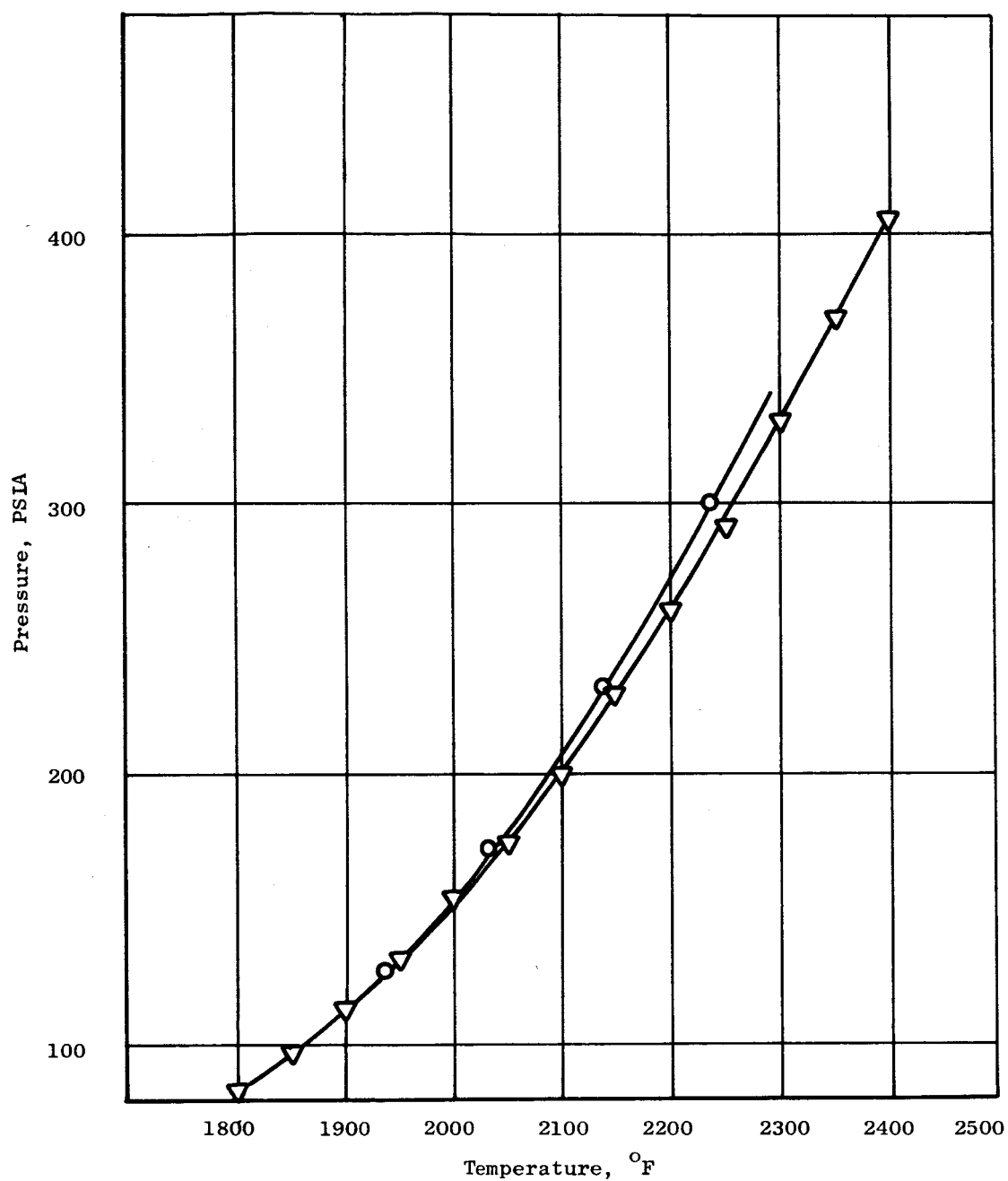


Figure 1. Estimated Average 1% Creep Properties of D-43 Alloy.



Data Source: ○ T. Phillips and M. McCarthy, Battelle Memorial Institute, January 7, 1964

▽ 12th Program Report, "High Temperature Properties of Sodium and Potassium", NRL6094, June 9, 1964

Figure 2. Vapor Pressure of Potassium.

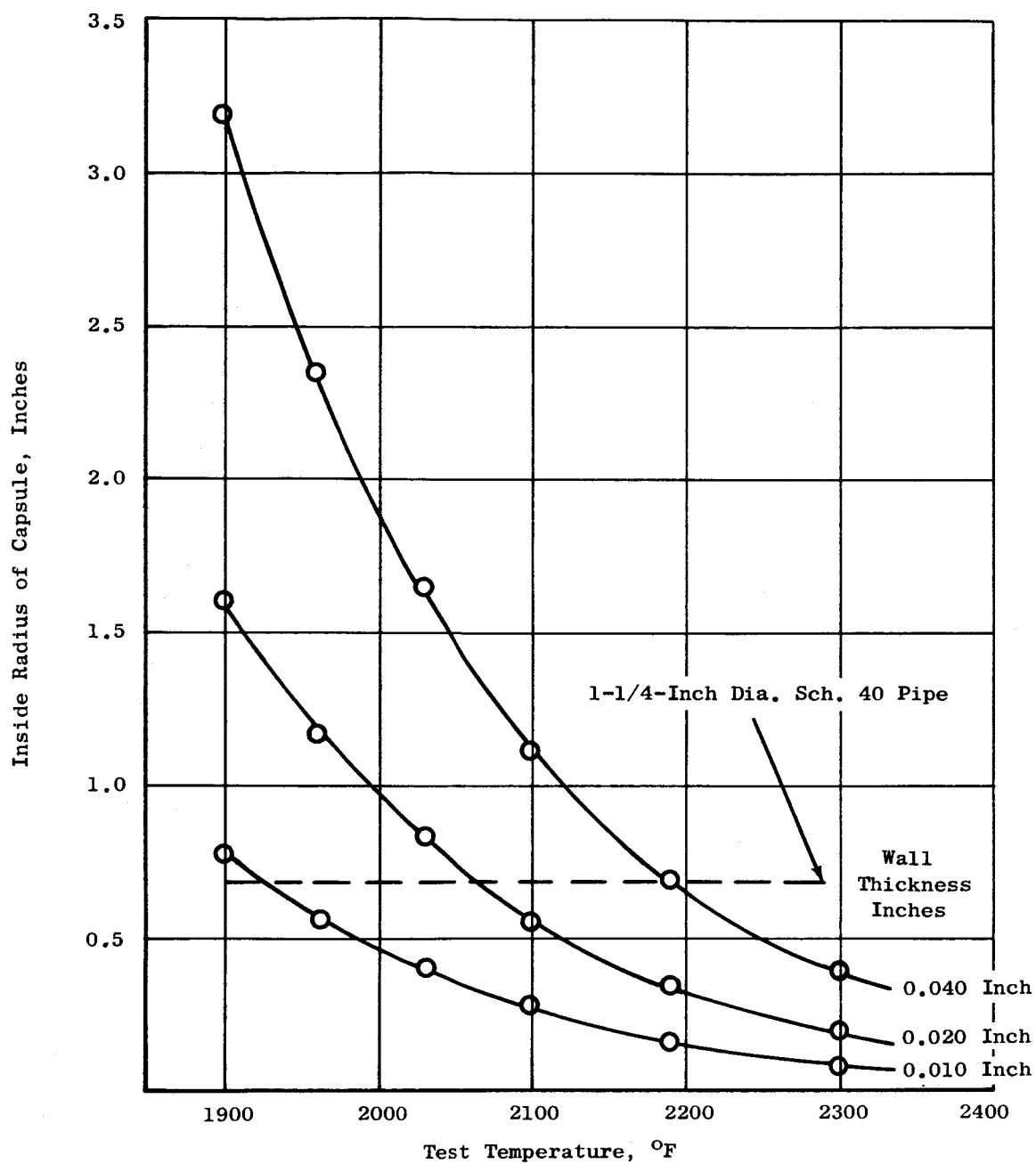


Figure 3. The Relationship Between the Test Temperature and the D-43 Alloy Capsule Radius to Produce 1% Creep in 300 Hours for Various Wall Thicknesses.

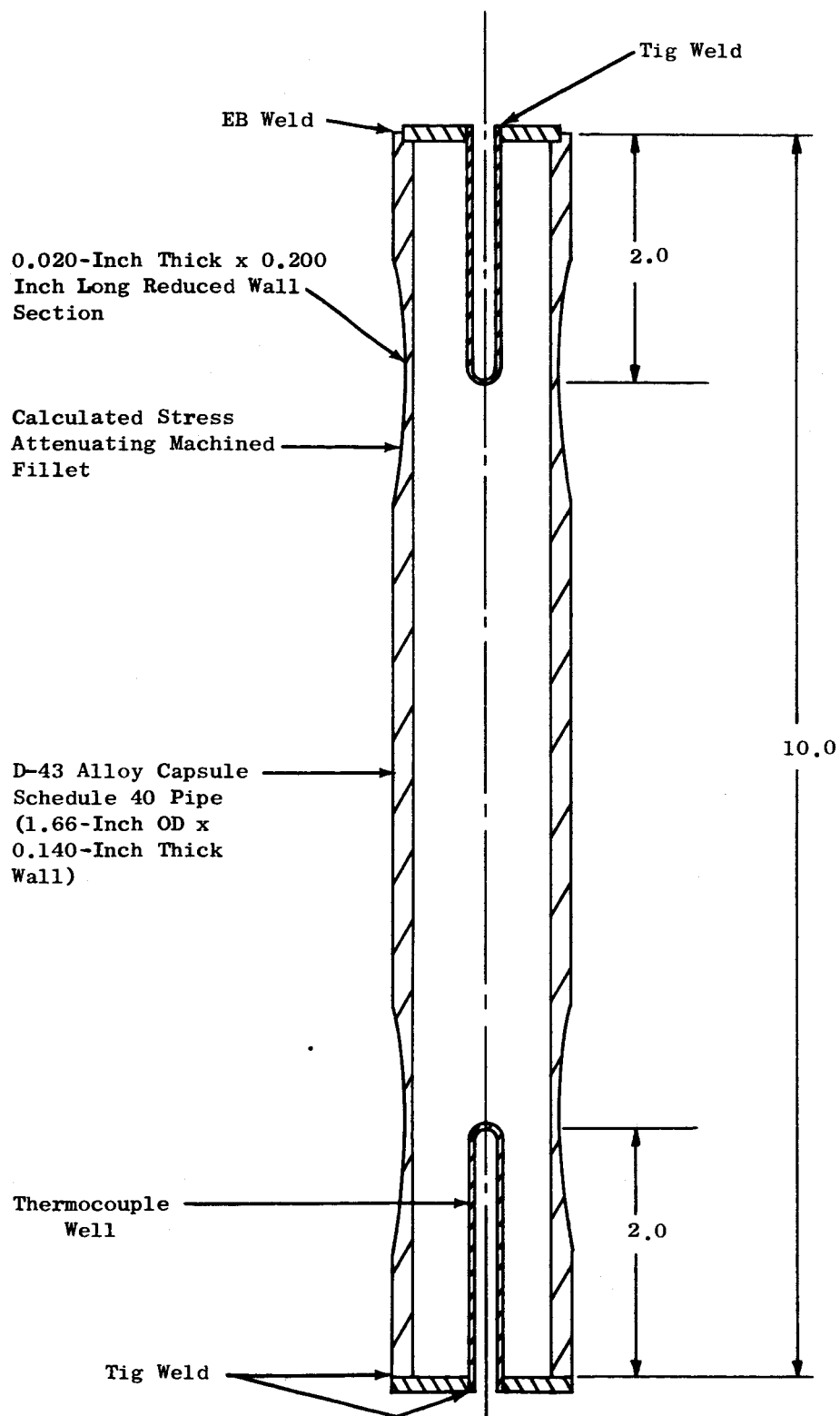


Figure 4. D-43 Alloy Refluxing Potassium Corrosion Capsule with Reduced Wall Sections for Measuring Induced Strain.

Thermocouple wells are located in the top and bottom of the capsules such that temperature measurements can be made in the boiling and condensing zones adjacent to the reduced wall sections.

C. Test Facility

The stress corrosion reflux capsule tests will be performed in a Varian high vacuum system (Model VI-16), Figure 5. The vacuum chamber, 18 inches in diameter and 30 inches high, is made of Type 304SS. The unit is bakeable to 450°C and is provided with two 4-inch diameter Pyrex windows and 11 feed-throughs. The pumping system consists of 3 cryogenic molecular sieve type roughing pumps and a 400 l/sec getter-ion pump that is capable of attaining a pressure in the 10^{-10} torr range in the chamber. The pressure is measured with a Hastings DV-6 thermocouple gauge from 1 to 1000 microns and a Varian 971-0003 Bayard-Alpert ionization gauge to 10^{-11} torr.

The capsules will be heated by tantalum strip heaters adequately shielded from the chamber walls with tantalum radiation shielding. The regulated voltage supply used to control the temperature is expected to maintain an accuracy of $\pm 1\%$ at 1000°F and above and $\pm 10^\circ\text{F}$ below 1000°F. Before initiation of testing, a complete checkout of the facility will be conducted. The temperature tolerances stated above are expected to be maintained at the test temperature and at pressures less than 1×10^{-7} torr.

The change in diameter in the thin-walled regions will be measured using high purity (99.5%) Al_2O_3 probes in conjunction with linear-variable-differential-transformers specially designed for high vacuum operation.

The design of the LVDT arrangement, shown schematically in Figure 6, has been completed; fixturing apparatus is being fabricated from Type 304SS.

D. Test Program

The capsules will be filled with high-purity potassium under a vacuum of less than 5×10^{-5} torr. Subsequently, the tops of the capsules will be sealed immediately in the vacuum by electron beam welding techniques. The potassium used is procured from Mine, Safety and Appliance Research Corporation as their high-purity grade and is further purified at GE by vacuum distillation at approximately 600°F at a pressure in the 10^{-3} torr range and hot trapping approximately 50 hours at

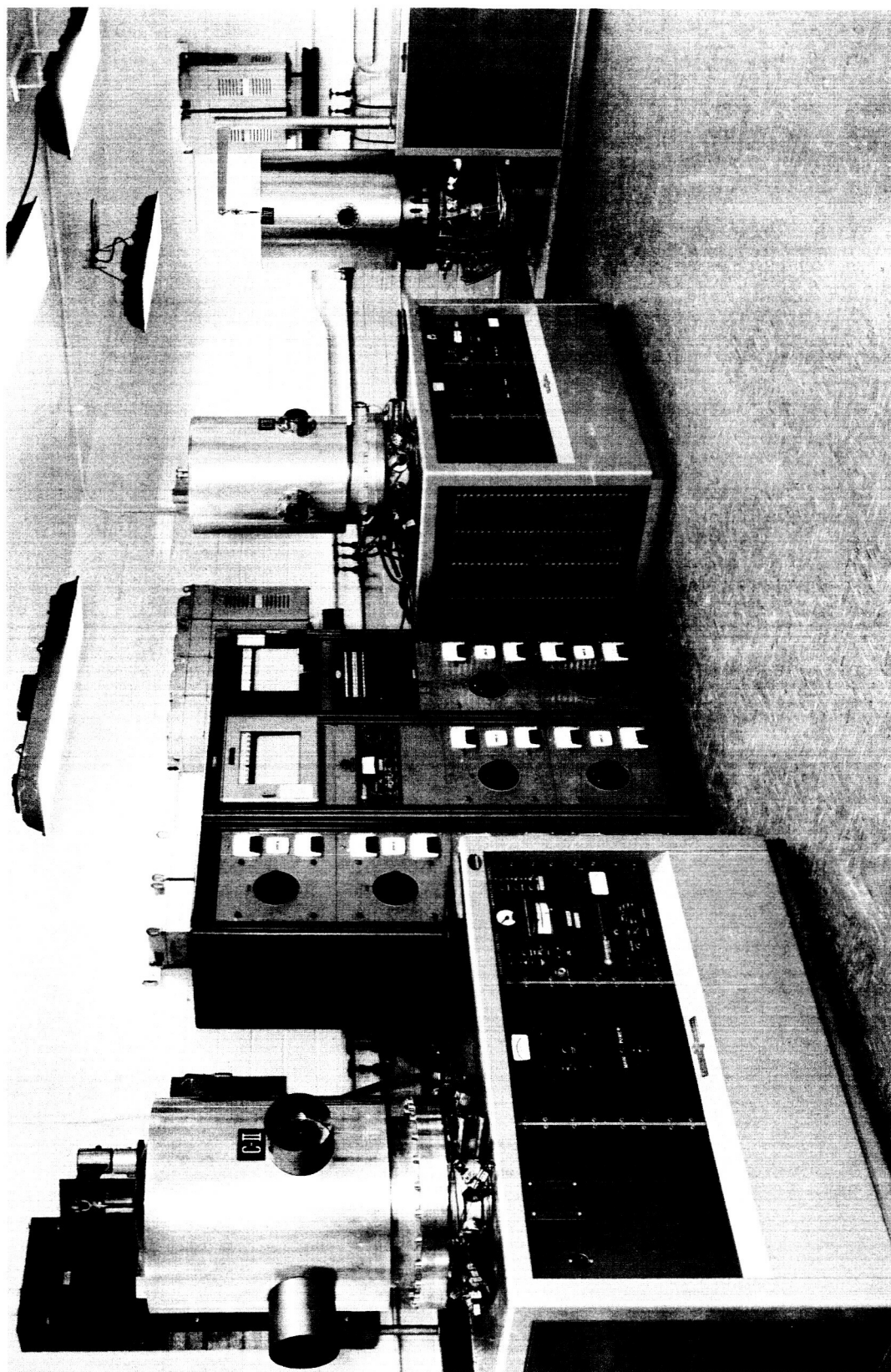


Figure 5. High-Vacuum System (10^{-10} Torr Range) (Middle) to be Used in the Evaluation of the Influence of Stress on the Corrosion Behavior of D-43 Alloy in Potassium in the Temperature Range 2000° to 2200°F . The Chamber, 18 Inches in Diameter and 30 Inches High, Incorporates a 400 X/Sec Getter-Ion Pumping System. (C64051216)

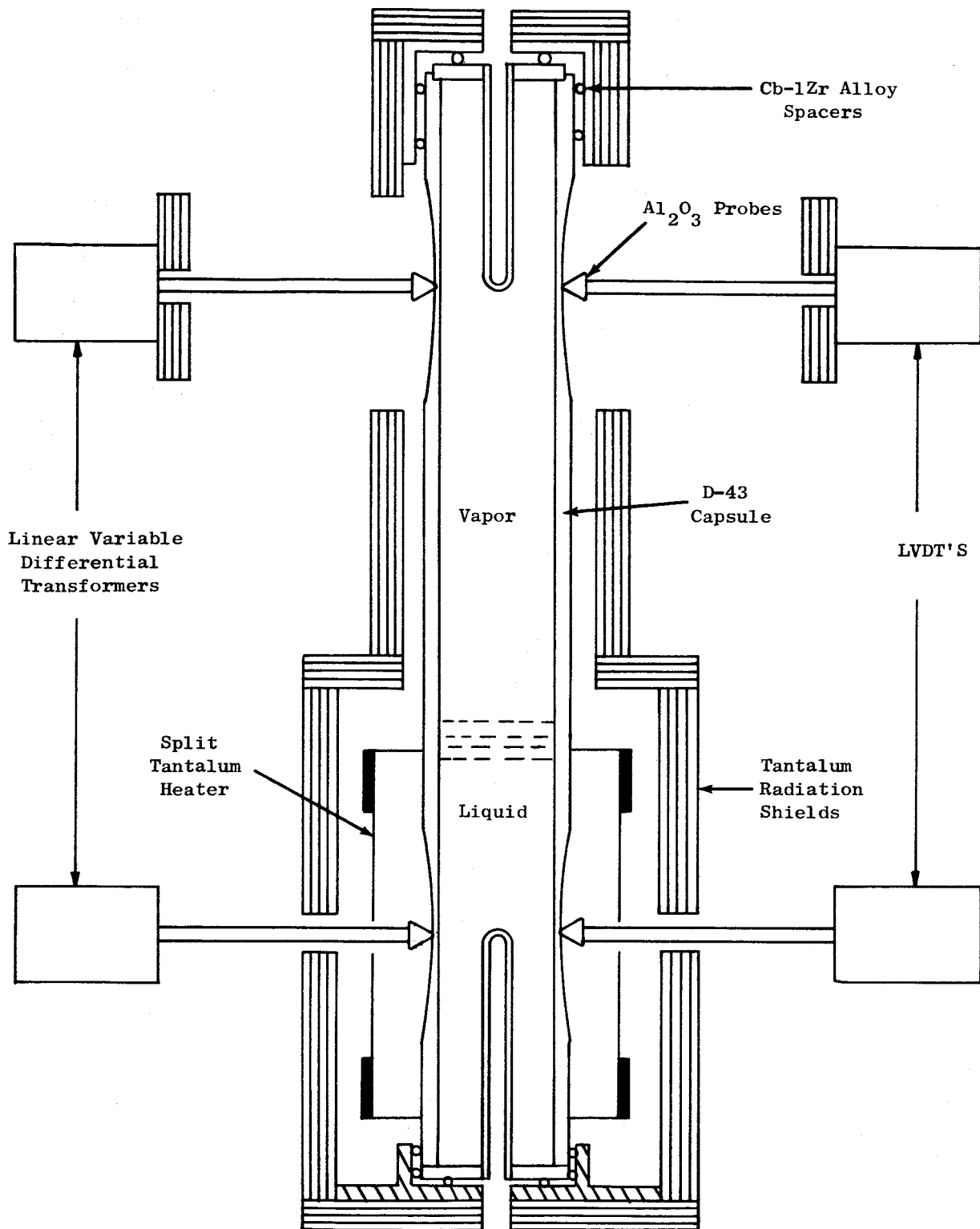


Figure 6. LVDT Arrangement for Measuring Induced Strain in the Thin-Wall Sections of Reflux Capsules.

1400°F in a titanium lined, zirconium gettered hot trap. The integral purification system is shown in Figure 7. Samples of the purified potassium will be obtained for the chemical analysis of metallic impurities by spectrographic techniques and for the analyses of oxygen by the mercury amalgamation method. Additional samples of the test potassium that are obtained during the filling operation shall also be analyzed for oxygen. After filling, the capsules will receive a careful visual and radiographic examination to assure their integrity prior to insertion in the high-vacuum chamber.

After the test exposures in the 2000°-2200°F temperature range, each of the capsules will be drained of the potassium under a helium environment and any residual potassium shall be removed by vacuum distillation. Particular attention shall be paid to determining the extent of carbon, oxygen, nitrogen and hydrogen mass transfer between the potassium liquid and vapor regions in the capsule by post-test analyses of the capsule wall sections. The pre-test and post-test evaluation of the capsules will include dimensional measurements, chemical analyses, electron microprobe analyses and metallographic examination.

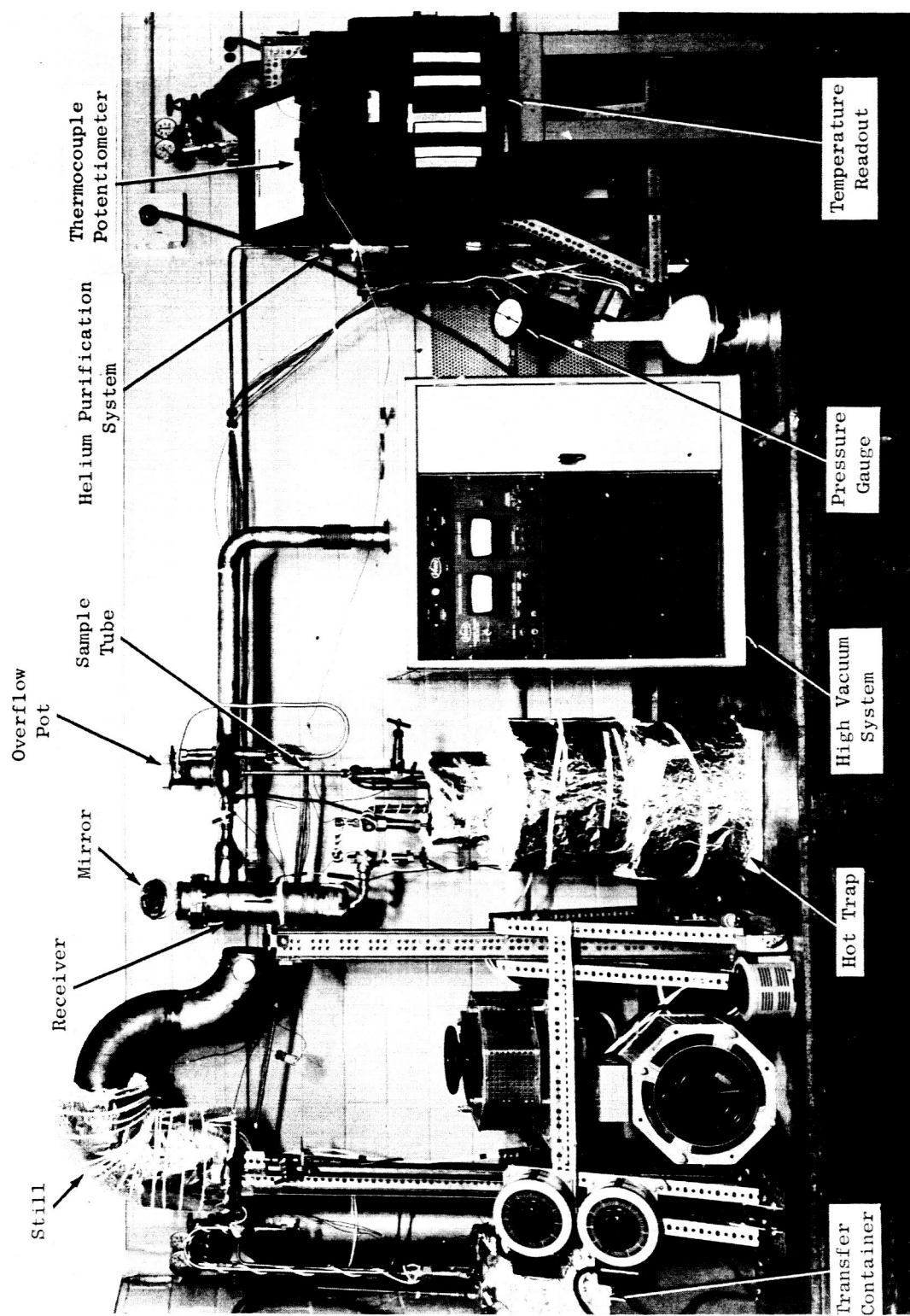


Figure 7. Potassium Purification System. (C64091641)

IV. TASK II - BIMETALLIC ISOTHERMAL CAPSULE TESTS

A. Materials Procurement

Capsules will be fabricated from 1-1/4-inch diameter, Schedule 80, Type 316SS and Type 321SS pipe as shown in Figure 8. The chemical analyses of the stainless steel pipe are shown in Table I and indicate that the titanium content in the Type 321SS is approximately seven times the carbon content. Since the molar ratio to form TiC requires the titanium content to be only 4 times the carbon content, the excess should insure that all the carbon and nitrogen atoms are combined as a carbide, nitride or carbonitride.

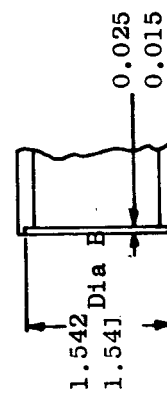
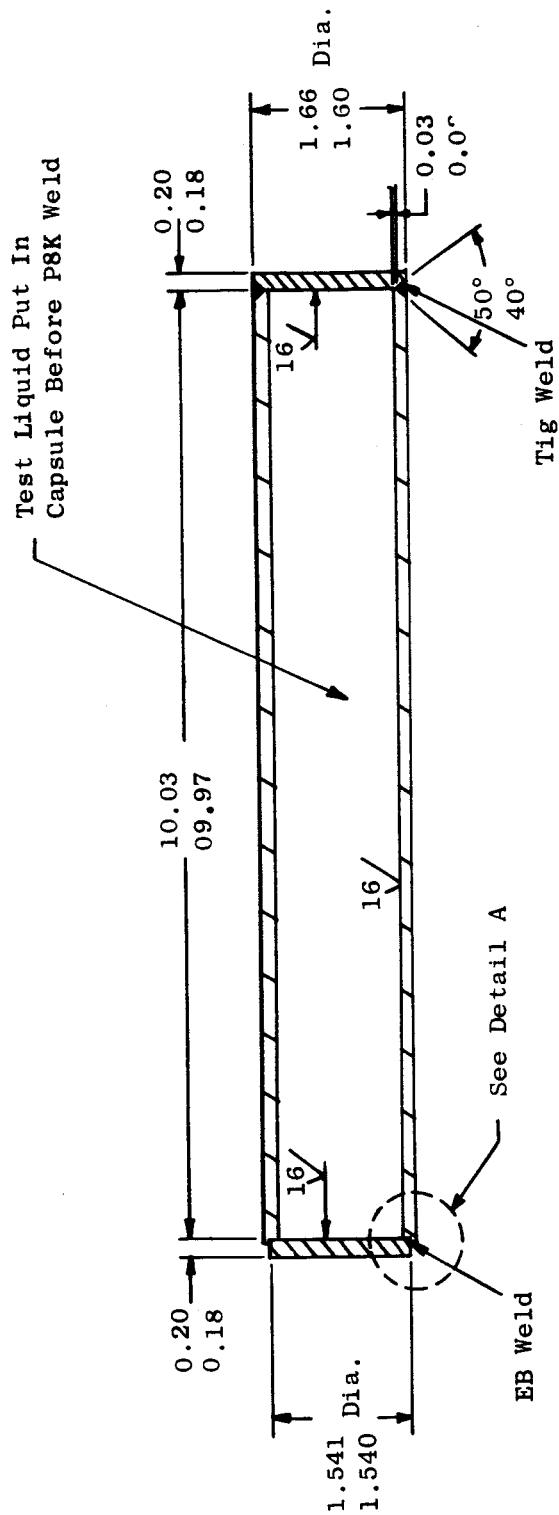
The Cb-1Zr alloy sheet that is to be contained within the capsules is on hand and tensile and stress-rupture sheet specimens (0.040-inch) are being machined, Figure 9. Chemical analysis of the material is shown in Table II.

B. Test Program

The stainless steel capsules will be tested isothermally for a period of 1,000 hours at approximately 1400°F in an air environment. The necessary heaters and instrumentation are on hand and are presently being assembled.

The potassium to be used for the capsule tests will be procured and purified in the same manner as described for Task I. The capsules will be filled under vacuum (10^{-5} torr range), using potassium that has been transferred directly from the final hot trapping container, and sealed by electron beam welding techniques. Samples of the purified potassium will be obtained for chemical analyses of metallic impurities by spectrographic techniques and of oxygen by the amalgamation method. Also, samples will be taken and analyzed for oxygen each time a set of capsules is filled.

After the test exposure, each of the capsules will be drained of the potassium under helium environment and the specimens cleaned by vacuum distillation. Evaluation of the Cb-1Zr alloy specimens will include dimensional and weight measurements, chemical analysis for oxygen, nitrogen, hydrogen and carbon, metallographic examination, tensile testing at room temperature and stress-rupture testing at about 2000°F. Evaluation of the stainless steel will include metallographic examination and chemical analysis for oxygen, nitrogen, hydrogen and carbon.



Detail A

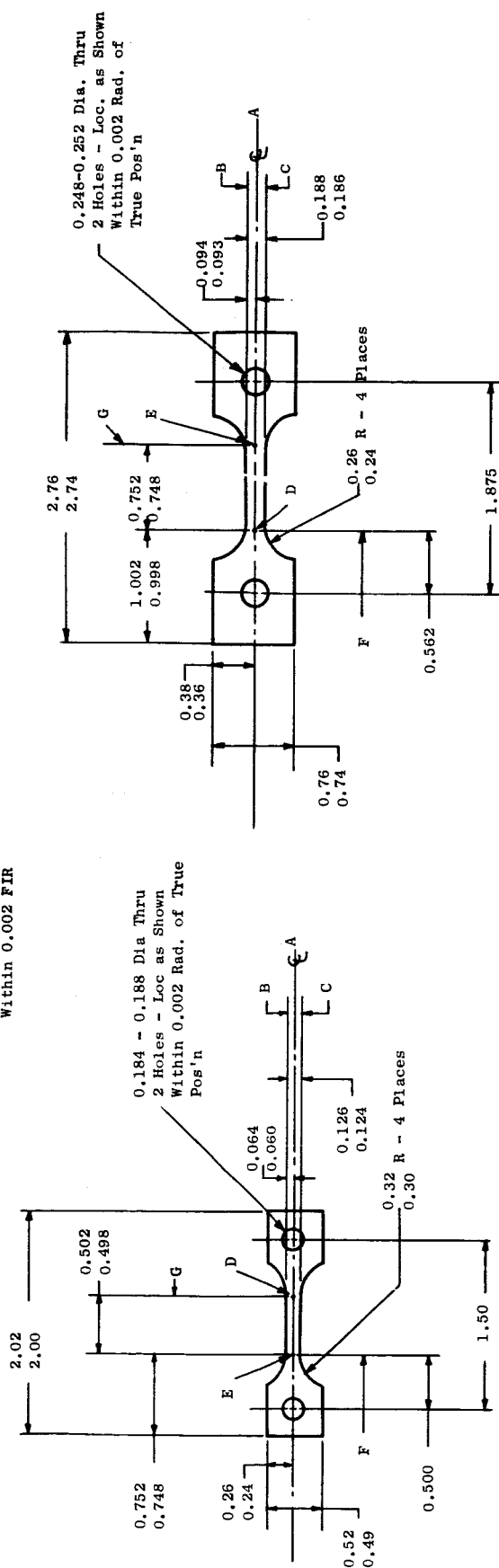
Figure 8. Stainless Steel Capsule Design.

TABLE I
CHEMICAL ANALYSIS OF 1-1/4-INCH DIAMETER
SCHEDULE 80 STAINLESS STEEL PIPE

<u>Element</u>	<u>Type 316SS,</u> <u>ppm</u>	<u>Type 321SS,</u> <u>ppm</u>
C	410	620
O	78	62
N	170	52
H	4	9
	<u>w/o</u>	<u>w/o</u>
Cr	17.39	17.34
Ni	12.33	11.51
Mg	1.86	1.66
Mo	2.24	<0.05
Ti	--	0.42
Si	0.75	0.61
Cu	0.21	0.09

Surfaces B and C are to be Parallel With Each Other and E Axis A Within 0.002 FIR

Planes F and G Must be Established Thru Points D and E and Perpendicular to Surfaces B and C Within 0.002 FIR



Sheet Tensile Specimen

Sheet Rupture Specimen

Figure 9. Tensile and Stress-Rupture Test Specimens.

TABLE II

CHEMICAL ANALYSIS OF Cb-1Zr ALLOY SHEET (MCN 454)

<u>Element</u>	<u>Vendor's Analysis⁽¹⁾</u> <u>ppm</u>	<u>GE Analysis</u> <u>ppm</u>
C	35	10
O	90	75
H	5	6
N	42	14
Zr	0.89%	-
Ta	500	-
Fe	50	-
Si	60	-
Hf	< 50	-
W	<100	-

(1) Kawecki Chemical Company

IV. FUTURE PLANS

A. Task I

1. Pre-test evaluation of the D-43 alloy will be initiated.
2. The capsule design will be completed.
3. The LVDT arrangement and fixturing device will be assembled.

B. Task II

1. Pre-test evaluation of the stainless steel pipe and Cb-1Zr alloy sheet will be completed.
2. Fabrication of the stainless steel capsules will be completed.
3. The facility design will be completed.
4. The heaters and necessary instrumentation will be assembled to perform the capsule tests.

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- (3) Grodzinski, P., "Investigation on Shaft Fillets," Engineering (London), Vol. 152 (1941), pp 321.

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